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PREPARATION OF THE SUPERCONDUCTOR SUBSTRATE: STRONTIUM TITANATE

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Preparation of the superconductor substrate, strontium titanate, is described. Procedures used to prepare the substrates for the strontium titanate boules are detailed. The strontium titanate crystals are grown using an oxidizing flame (hydrogen to oxygen ratio of 1). The other parameters used for crystals growth are reported. The growth direction was determined to be 5 degrees away from the [211] direction. ICP-emission spectroscopy indicates that the burner material may contribute a small amount of impurities to the crystals.

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Preface

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Preparation of the Superconductor Substrate: Strontium Titanate

1. INTRODUCTION

Ceramic superconductors with transition temperatures (Tc's) above the boiling point of liquid nitrogen (>77K) have received an unprecedented amount of investigation since their recent discovery.[1,2,3] These superconductive materials have been predicted to revolutionize transportation, electrical transmission, magnetic instrumentation and microcircuitry. The immediate application of these superconductors will most likely be realized in the area of microcircuitry such as computer interconnects, semiconductor-superconductor hybrids, Josephson junctions, etc.

The application of superconductors in microelectronics necessitates the development of techniques to prepare thin films of the superconductive materials. Various approaches for the preparation of the high Tc superconducting films are currently underway, such as electron beam evaporation, single target magnetron sputtering, magnetron sputtering from three metal targets, laser deposition, molecular beam deposition, etc. Thin film formation requires substrates that are

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compatible with the superconductor being deposited. Among the properties that need to be compatible are (1) the same crystal structure with matched-lattice constants, (2) similar thermal expansion and (3) minimum interface reactions. Strontium titanate has been shown by various investigators to exhibit these characteristics required for their use as substrates for the superconductive films.

A program was initiated to investigate the synthesis of crystalline strontium titanate. Crystals of strontium titanate have been grown by various techniques: flame-fusion[4,5,6,7], flux-growth[8,9,10,11,12], floating zone[13], Czochralski method[14] and solution growth.[15] Among the techniques, the flame-fusion method has the advantage of producing large size single crystals. Wafers of appreciable dimensions are required when used as substrates for the superconductive thin films. This report describes the synthesis of single crystals of strontium titanate with the flame-fusion technique and their fabrication to superconductor substrates.

2. EXPERIMENTAL

The flame-fusion techniques for the growth of single crystals of strontium titanate is derived from the original method developed by Verneuil.[16] The general procedure for the growth of single crystals by the flame-fusion technique is depicted in Figure 1. A highly purified feed-powder of strontium titanate is tapped through a 40-mesh sieve into a stream of oxygen flowing in the center

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4. Bednorz, J.G. and Scheel, H.J. (1977) Flame-fusion growth of $SrTiO_3$, *J. Crystal Growth* **41**:5.
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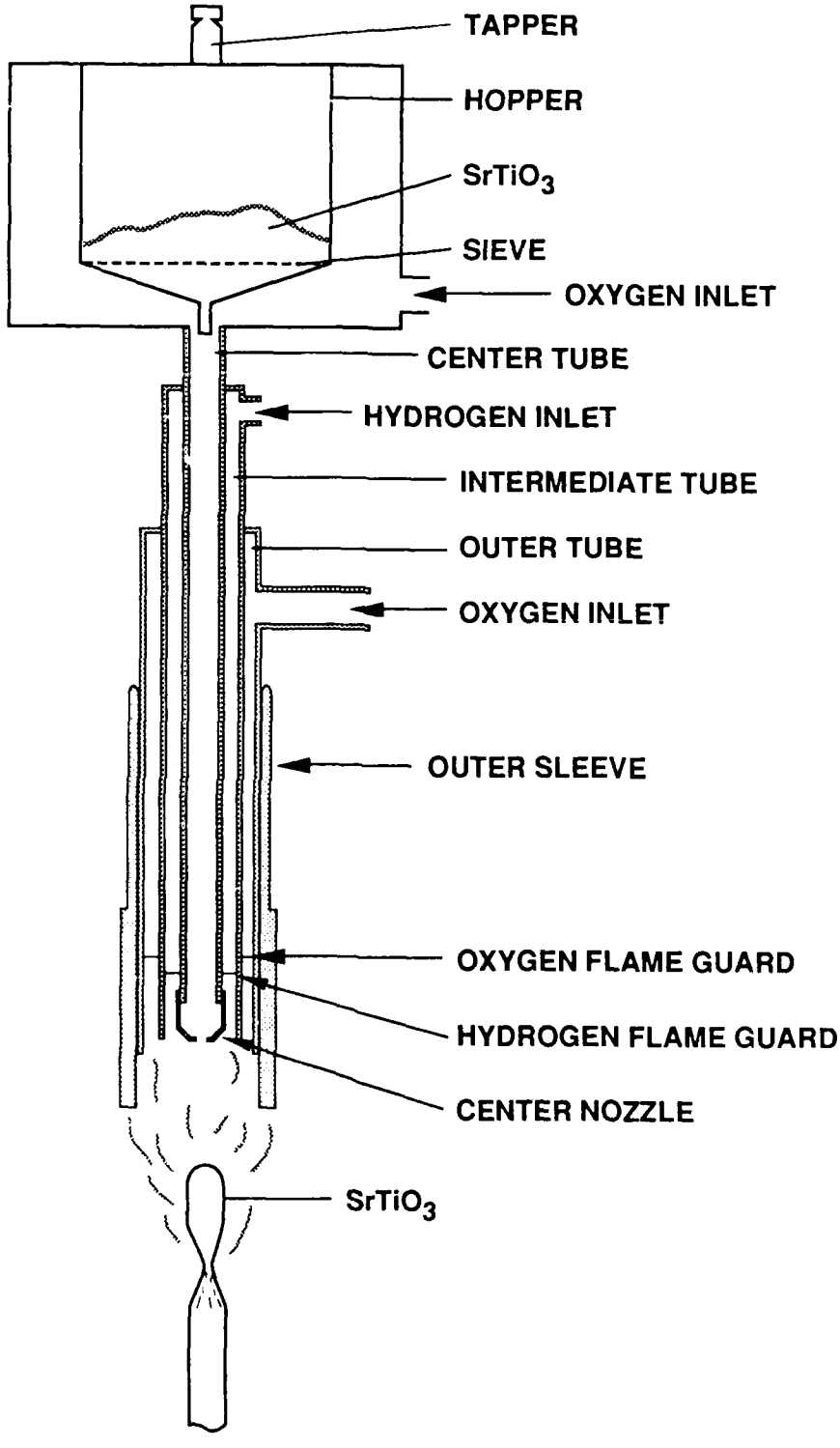

Figure 1. Schematic Diagram of the Flame-Fusion Apparatus

tube of an oxy-hydrogen burner. The burner is a three-tube postmix type with the center tube carrying oxygen; the intermediate tube, hydrogen; and the outer tube, oxygen. A detailed description of the burner is presented in the burner section of the report. The flame of the burner is directed into a 3.8 cm top-center hole of a refractory sleeve prepared from cast alumina. The refractory sleeve is 25.4 cm in diameter and 24.1 cm high. The sleeve contains a side-view circular window (diameter, 1.9 cm) whose center is located 7.3 cm from the top of the refractory sleeve. The center of the window is in line with that temperature of the flame where the $SrTiO_3$ crystal is grown. Bednorz et al[4] reported the growth of the single crystal of strontium titanate in the range of 2470-2480° C. In the initial segment of growth, the feed powder falls on a pressed-alumina pedestal whose position is at a temperature where a sintered cone of $SrTiO_3$ is formed. The top of the sintered cone is then positioned at that temperature where a small melt droplet of $SrTiO_3$ is formed. A neck of approximately 5 mm is formed by lowering the pedestal at a relatively high rate. The diameter of the crystal is then increased by lowering the pull-rate of the pedestal. Boules of strontium titanate 6 cm long and 1.4 cm in diameter have been achieved by this method. Single crystals may also be grown from seeds.

2.1 Three-Tube Postmix Burner

The development of the burner has been adequately described by J. Adamski.[17] The burner consists of three stainless steel concentric tubes of different diameters. The center tube carrying the oxygen and feed powder is threaded at the bottom so that different inconel nozzles may be used. The intermediate tube, which carries the hydrogen, contains an inconel baffle with a series of holes permitting the concentric flow of hydrogen. The holes are equally spaced with diameters of 0.062 inch on a 0.531 inch diameter bolt circle. The center tube and the intermediate are held together by a specially constructed T made of brass. The outer tube carrying the oxygen is silver soldered to the intermediate tube. The outer tube contains an inconel baffle at its base. The baffle contains a series of holes (0.062 inch diameter, equally spaced on a 0.937 inch bolt circle) permitting the flow of oxygen. A water-cooled inconel sleeve is placed over the outer tube of the burner. This sleeve may be positioned along the outer tube until a desired flame pattern is obtained.

2.2 Preparation of Strontium Titanate Feed Powder

The preparation of the feed powder is critical. The powder must be relatively free of impurities and possess a certain particle size. Bednorz et al.[4] and Linz et al.[5] used particle sizes in the range of 0.05-0.125 mm and 0.05-0.74 mm, respectively, while Merker[6] reported the use of particle sizes less than 0.149 mm. The method to prepare the feed powder in the present study was the oxalate precipitation procedure modified after Linz et al.[5]

Oxalic acid ($C_2H_4O_4 \cdot 2H_2O$, 177g) was dissolved in 370 ml of distilled water in a jacketed beaker. The solution was maintained at 70° C by means of a constant temperature bath with a circulating pump. A solution containing 40 ml of titanium tetrachloride in 121 ml of distilled water was then added to the oxalic acid solution. The titanium tetrachloride solution was prepared by adding $TiCL_4$

17. Adamski, J.A. (1956) New oxy-hydrogen burner for flame fusion, *J. Appl. Phys.* **36:**1784.

(99.9%) to distilled water in a beaker that was cooled by an ice bath. The oxalic acid solution containing the $TiCl_4$ was then equilibrated to 70° C. A solution containing 152.0g of $SrCl_2 \cdot 6H_2O$ in 590 ml of distilled water was added rapidly to the oxalic acid-titanium tetrachloride solution. Slow addition resulted in powder having a particle size greater than 0.131 mm. Upon the addition of the strontium chloride solution, a white precipitate formed. This precipitate was digested for two hours at 70° C. The precipitate, filtered by suction, was washed with 8 liters of distilled water. The precipitate was dried overnight by suction.

We added 2.1g $SrCO_3$ to the partially dried precipitate. Bednorz et al.[4] reported that an excess of SrO (equivalent to 3.8 percent $SrCO_3$) is necessary to compensate for the SrO lost in the flame-fusion process. A deficiency in strontium oxide produces a melt enriched in titanium oxide which leads to an overflow of the molten cap on the boule. The amount of $SrCO_3$ added in the present procedure is equivalent to the final powder of $SrTiO_3$ containing 3.8 percent of the carbonate. This calculation is based on an 80 percent yield determined previously for this $SrTiO_3$ process. The strontium titanate precipitate was transferred to zirconia crucibles and dried in a drying oven. The precipitate was slowly dried to 175°C and then for two hours at 1000°C in a muffle furnace. Analysis calculated for $SrTiO_3$: Sr, 47.7 percent; Ti, 26.1 percent. Found: Sr, 47.8 percent; Ti, 25.6 percent. Analyses for impurities by ICP-emission spectroscopy found: Ca, < 0.01 percent; Si, < 0.01 percent; Cr, 0.01 percent; Fe, 0.09 percent; and Al, 0.03 percent.

The dried $SrTiO_3$ powder was sieved using a nest of three sieves: 200, 270 and 325 mesh. The sieve analysis of the feed powder is depicted in Figure 2. The figure shows that 91.3 percent of the feed powder has a particle size of less than 0.044 mm. This size fraction was used in the preparation of the strontium titanate crystals.

2.3 Growth of Strontium Titanate Crystals

The operational parameters of a typical run for the growth of a $SrTiO_3$ boule from a cone are given in Figure 3. The initial tap rate was 40 taps/min and was decreased to 24 taps/min after 15 minutes. The oxygen flow rates were constant at 15 l/min and 5 l/min for the outer and inner tubes, respectively. The hydrogen flow rate was increased from an initial value of 13.5 l/min to 21.0 l/min. The sintered cone was formed at the lower flow rate (lower temperature). Growth was carried out in an oxidizing flame where the ratio of H_2/O_2 was 1. This is in agreement with Gorina et al.[18] who used an oxidizing flame to obtain clear transparent crystals. Bednorz et al.[4] reported a slightly reducing flame where the H_2/O_2 ratio was 2. Merker [6] used a strongly reducing flame with H_2/O_2 equal to 10. These results indicate that either a reducing or an oxidizing flame is capable of producing high quality single crystals of $SrTiO_3$. A boule approximately 4 cm long and 1.5 cm in diameter was produced with these operational parameters. A large portion of the boule was single crystal.

The $SrTiO_3$ boules were annealed in zirconia crucibles. The boules were covered with $SrTiO_3$ feed powder and heated at 1000°C for 24 hours. The boules turned from black to colorless. Some of the

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18. Gorina, Yu. I., Kashtanova, A.M., Maksimova, G.V., and Skanavi G.I. (1961) Preparation of monocrystals of strontium titanate and some data on their dielectric properties, *Kristallografiya* **6**:473.

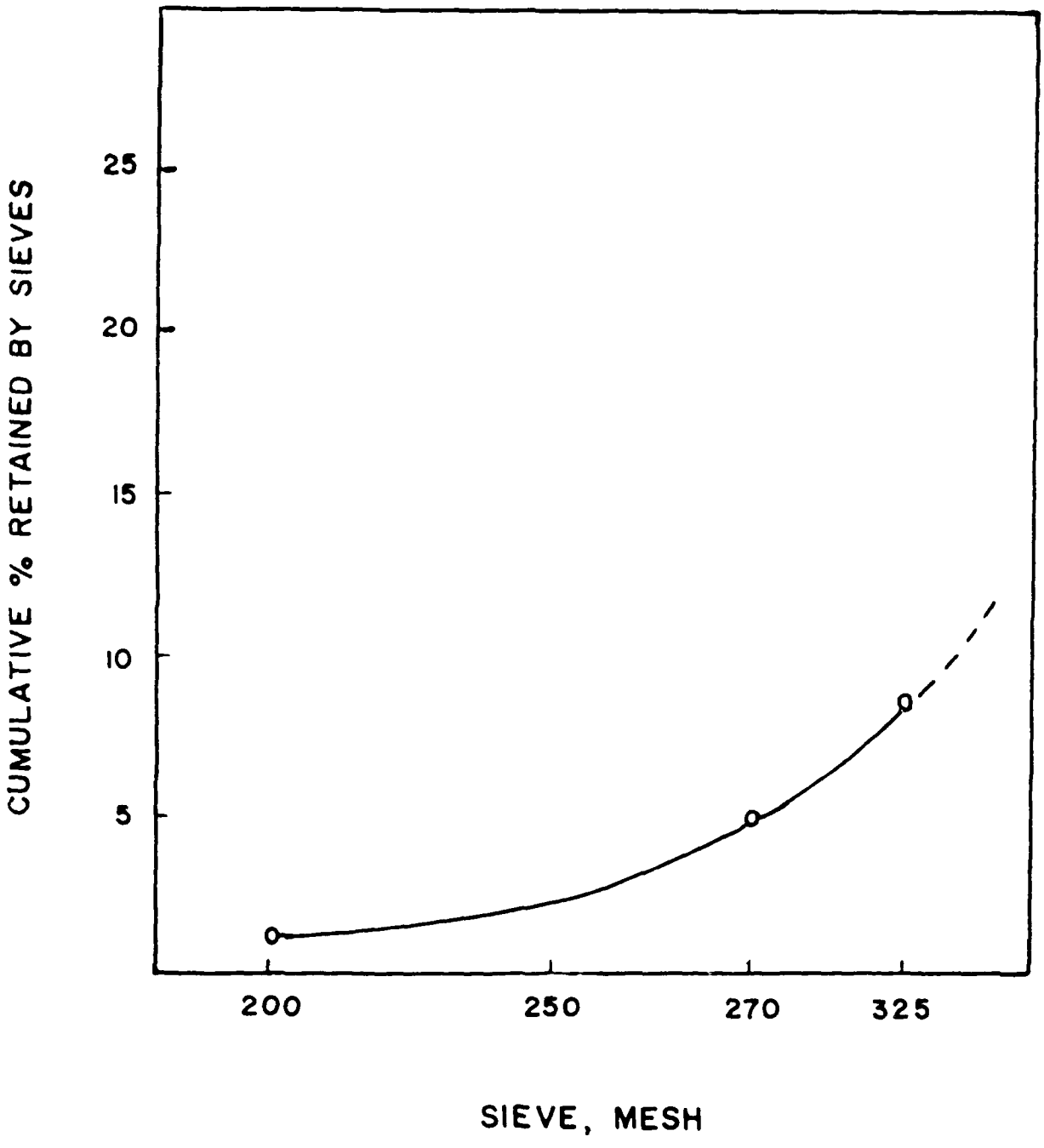

Figure 2. Sieve Analysis of the Strontium Titanate Feed Powder

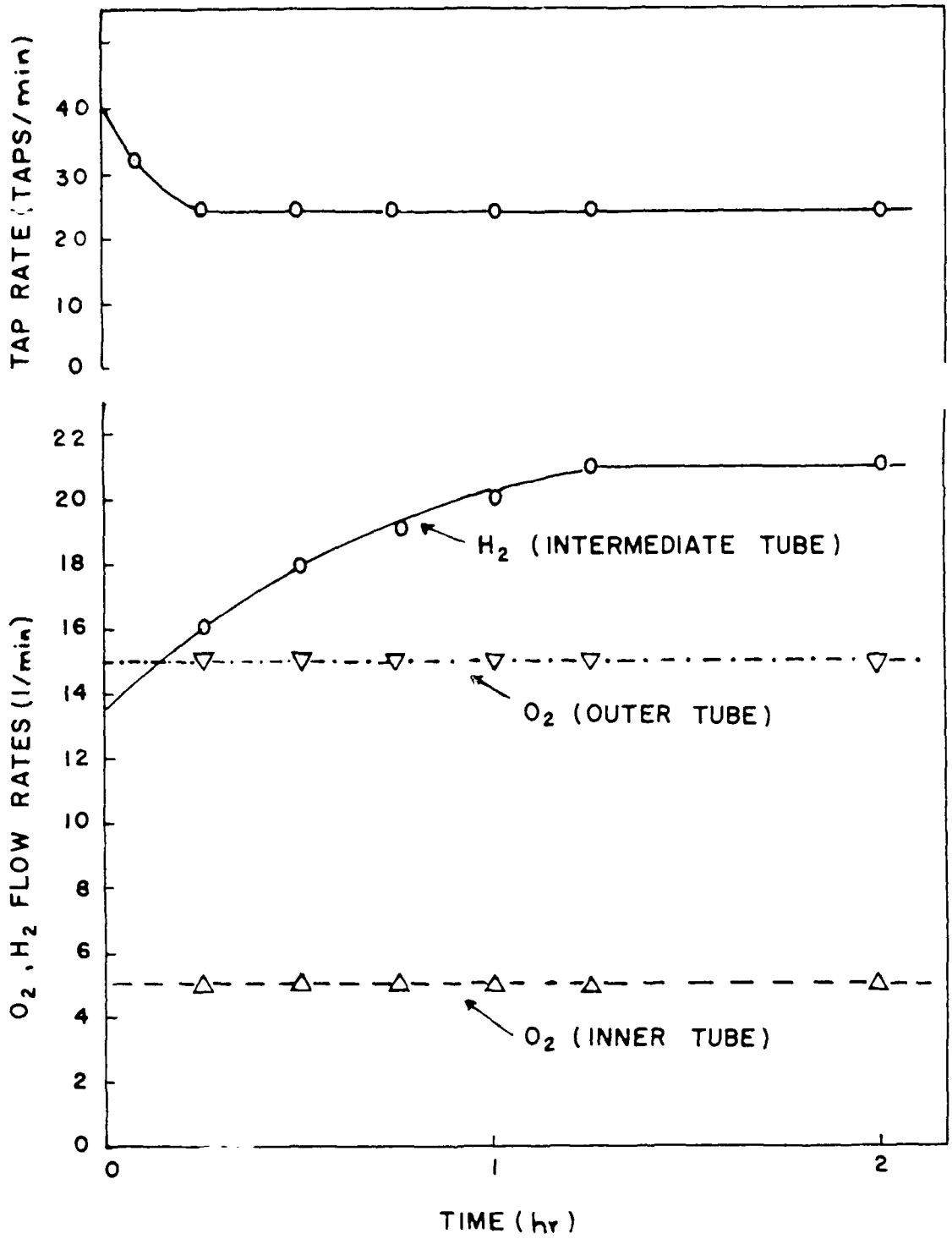

Figure 3. Operational Parameters Used for the Growth of $SrTiO_3$ Crystals by the Flame-Fusion Technique

boules possessed a yellowish tint. The black coloration may be due to a deficiency of oxygen in the crystals.

2.4 Preparation of The Substrates

Wafers of strontium titanate have been shown to be ideal substrates for the thin films of the superconductive ceramics.[19,20,21] Therefore the boules must be sliced and polished. The $SrTiO_3$ boules were sliced using a diamond-coated metal blade. The diamond was 220 grit at a concentration of 75 percent at a 1/8 inch depth. Water was the coolant in the slicing procedure.

The polishing consisted of a rough grind with 320 grit SiC paper or SiC on glass plate to obtain a smooth planar surface. The slices were further polished with successive treatments of 15, 9, and 6 micron microgrit (Al_2O_3) with oil on a glass plate. This same treatment was repeated on a cast iron wheel on a bowl polisher. The final polishing was accomplished with 1 micron colloidal SiO_2 paste on nylon cloth or on politex supreme pads.

3. RESULTS AND DISCUSSION

Figure 4 shows a polished segment of the strontium titanate crystal obtained by the flame-fusion technique. The photograph demonstrates the excellent quality of the crystal. This quality is further depicted in the SEM micrograph of the surface of the $SrTiO_3$ crystal. These results show that crack- and inclusion-free crystals can routinely be obtained with the flame-fusion technique. The lattice constant (a) determined from x-ray diffraction studies was found to be 3.9056 ± 0.0006 Å. This is in excellent agreement with the value of 3.9050 Å reported by Swanson et al.[22]. Laue back-reflection photographs of the polished surface showed that the growth direction was 5° off the [211]. This result differs from Linz et al.[5] who suggested that a preferred growth direction is observed along the [110]. Bednorz et al.[4] studied the growth directions of 20 crystals and indicated no preferred growth orientation. Spot splitting in the present study indicated the presence of low angle grain boundaries.

Some crystals possess a slight yellowish tint, possibly due to the presence of a trace amount of iron. An ICP-emission spectroscopy analysis showed the presence of the following impurities: Ca, 0.03 percent; Si, <0.01 percent; Fe, 0.03 percent; Al, 0.06 percent; Mo, 0.17 percent; and Co, 0.04 percent. The presence of molybdenum and cobalt in the crystal is probably due to contamination by the burner material since these elements are not found in the feed powder. Bednorz et al.[4] reported

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Figure 4. Polished Segment of a Strontium Titanate Crystal Grown by the Flame-Fusion Technique

Figure 5. SEM Micrograph of the Polished Surface of a Strontium Titanate Crystal (4000X)

their strontium titanate crystals contained 0.005 percent Fe^{+3} and Cr^{+3}, but indicated that crystals prepared from high-purity strontium titanate feed powder contained only a few ppm of Fe^{+3} (~0.0003 percent). The crystals prepared by Gorina et al.[18] contained the following impurities: Mg, 0.006 percent; Si, 0.006 percent; Al, 0.01 percent; Fe, 0.003 percent. The differences in percentages found for the impurities in the different studies may possibly be due to the sensitivity of the various methods of analyses such as EPR, spectral analysis and ICP-emission spectroscopy. Mo (VI) does not exhibit an electron spin resonance spectrum.

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